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THE EFFECT OF POST-CURE AND  
ANTIMONY TRIOXIDE ADDITION ON  
THE GLASS TRANSITION OF POLYESTER AND  
VINYL ESTER RESIN SYSTEMS

R. M. Morchat - I.A. Keough - J.G. Dwyer

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Approved by R.T. Schmitke  
Director/Technical Division

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## ABSTRACT

The effect of post-curing on the glass transition temperature ( $T_g$ ) of a variety of styrene cross-linked polyester and vinyl ester resins was determined by monitoring the changes to  $T_g$  using a Differential Scanning Calorimeter. The effect of adding the inorganic fire-retardant, antimony trioxide, on the  $T_g$  of these resin systems was also investigated.

Results indicated that the effect of post-curing was to increase the amount of cross-linking of the polyester and vinyl ester resins, as evident by the beneficial increase in the glass transition temperature and changes to the storage modulus. The addition of up to 10 parts per hundred resin (phr) of the fire-retardant, antimony trioxide, had a negligible effect on the onset of the glass transition point.

## RÉSUMÉ

Le contrôle des variations de la température de transition vitreuse ( $T_g$ ) par analyse calorimétrique différentielle à compensation de puissance a permis de déterminer les effets du post-traitement sur la  $T_g$  de plusieurs résines de polyester et d'ester vinylique réticulées par le styrène. Le présent document explore aussi les effets de l'addition de trioxyde d'antimoine, un produit ignifuge inorganique, sur la  $T_g$  de ces systèmes de résines.

Les résultats de l'étude indiquent que le post-traitement a pour effet d'augmenter le degré de réticulation des résines de polyester et d'ester vinylique, comme en font foi l'augmentation de la température de transition vitreuse et les variations du module de conservation. Cependant, l'addition jusqu'à 10% de trioxyde d'antimoine a un effet négligeable sur la température de transition vitreuse.

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## NOTATION

°C	Degree Celsius
deg	Degree
DMA	Dynamic Mechanical Analysis
DSC	Differential Scanning Calorimeter
E'	Storage Modulus
GPa	Gigapascal
GRP	Glass Reinforced Plastic
HPSEC	High Performance Size Exclusion Chromatography
min	Minute
mcal	Millicalories
mL	Milliliter
MW	Molecular Weight
PHR	Parts Per Hundred Resin
sec	Second
TGA	Thermal Gravimetric Analysis
T <sub>g</sub>	Glass Transition Temperature
T <sub>min</sub>	Peak Minimum Temperature
T <sub>post-cure</sub>	Post-Cure Temperature
ν	Cross-Link Density

## 1. INTRODUCTION

Reinforced polymeric materials, in particular glass reinforced plastics (GRP's), are finding increased utilization in such defence applications as sonar domes, submarine casings and minesweeper hulls. In a marine environment, GRP materials offer potential advantages over traditional construction materials in terms of significant weight savings due to their high strength to weight characteristics, and low maintenance due to the elimination of corrosion; however, the fire performance properties of these polymeric materials continue to be a major concern, limiting their use in naval vessels. The susceptibility of a naval warship to damage from fire and associated smoke dictates that any polymeric material considered for use should be as fire resistant as possible and be a low smoke producer.

Reliability of these materials under service conditions is another major concern. An understanding of the physical behavior, including the chemical, mechanical, and thermal properties of glass reinforced plastics, requires that the resin, the reinforcing fibre, the additives (such as fire retardants), and their interaction must be fully understood. The aim of an ongoing research effort is to optimize the fire performance and physical properties of GRP's [1].

Polyester and vinyl ester thermoset resins reinforced with 'E'-glass fibres are materials of interest for use as GRP composites in a marine environment. The molecular structure of thermosetting resins is a highly cross-linked network that is stable in heat and cannot be made to flow or melt. This network is constructed by the interconnection of smaller molecules, such as styrene, during a process called curing. During the curing process, increasing the temperature provides the resin with more energy and the

curing proceeds at a faster rate. Thus over the same amount of time, the amount or degree-of-cure increases, with increasing cure temperature, up to the point when curing is complete. Beyond the temperature at which complete curing takes place (the optimum curing temperature), decomposition of the resin will eventually occur.

The key property of a thermoset, which ultimately determines the level of all physical properties, is the degree-of-cure. The degree-of-cure can influence parameters such as the Young's modulus and thermal expansion coefficient, which affect the stability and mechanical integrity of the resin at elevated temperatures [2].

The degree-of-cure is also an important variable in the burning characteristics of thermoset polymers. Increasing the degree-of-cure generally increases the fire retardancy, probably due to the decrease in the amount of volatile combustion products as the cross-linked density is increased. It is for this reason that polymers must be fully cured before being submitted to any burning tests.

Differential Scanning Calorimetry is an analytical technique which can be used to monitor the heat capacity of a polymer and allow for measurement of the exothermic process of curing and the endothermic glass transition peaks.

The glass transition phenomenon is one of the most important properties of a polymer [3]. The glass transition temperature ( $T_g$ ) of polymers is closely connected with the occurrence of internal stress, the cohesive energy density, the mechanical property and the cross-link density. The higher the  $T_g$ , the higher the temperature at which the polymer is useful in structural applications.

Glass transition occurs at a temperature when there is enough heat energy available to increase the local molecular



motion of the resin enough to enable the resin to go from a brittle, glass-like state to a more flexible state. In theory, a more-interconnected resin network requires more energy to achieve the glass transition. In other words, the more the resin is cured, the higher the  $T_g$ . In fact, at the glass transition point dramatic changes in physical properties occur.

Another factor which can affect the physical properties is the addition of a fire-retardant. Unlike other additives, fire-retardants can appreciably impair the mechanical properties of a composite. Fire-retardants can have an adverse effect on the strength of the resin mixture and also on the bond strength between the resin and the fibre. Thus, an understanding is required of the effect the addition of an inorganic fire-retardant, such as antimony trioxide, has on the polymerization process.

## **2. EXPERIMENTAL PROCEDURE**

### **2.1. Resins Evaluated**

Five resins were evaluated in this study: Hetron 99P, Hetron 197AT, Hetron 27196, Hetron 692TP25 (Ashland Chemicals) and Derakane 510A (Dow Chemical Canada Inc). Information from the resin manufacturers' product data sheets indicated that: Hetron 99P is a fully promoted, thixotropic, fire-retardant, chemical resistant polyester resin; Hetron 197AT is a Class 1 fire-retardant, chemical resistant, heat resistant, unsaturated polyester resin; Hetron 27196 and Hetron 692TP25 are low viscosity, thixotropic, halogenated, flame retardant polyester resins containing styrene; and Derakane 510A is a corrosion resistant, chemical resistant, fire resistant vinyl ester resin. The resins were catalyzed with 0.15 phr (parts per hundred resin by weight) of the accelerator cobalt naphthenate (Nuodex DMR, Nuodex Canada

Ltd) and 1.0 phr of methyl ethyl ketone peroxide catalyst (Lupersol DDM-9, Pennwalt).

All of the resins used in this study contained proprietary halogenated materials which were intended to provide fire-retardancy characteristics through the chemical combination of chlorine and/or bromine molecules with the polymeric resin.

## **2.2. Sample Preparation**

Solid or liquid samples to be studied by Differential Scanning Calorimetry were placed in a sample pan and a sample pan cover was crimped in position using a crimper press. Tweezers were used for handling both the sample and the pans as body oils can produce spurious peaks. Maximum peak sharpness and resolution was obtained when surface contact between the sample and sample pan was optimum. Thus, sample configurations such as thin discs or films gave the best results.

Sample preparation of the resin systems involved mixing the resins with the required volume of catalyst and fire retardant and allowing the mixture to cure, at room temperature (20°C), in the aluminum sample pans. Aluminum pans were used for the analysis since the temperature range of interest was below the melting point for aluminum.

A pair of hardened samples of the different resin mixtures was then placed in a drying oven for one hour at either 50°C, 75°C, 100°C or 125°C. The temperature of the drying oven never deviated by more than  $\pm 2^\circ\text{C}$ .

## **2.3. Instrument Parameters**

The samples were analyzed on the Perkin-Elmer Model DSC-2 Differential Scanning Calorimeter [4] and all data were analyzed using the Perkin Elmer Model 3700 Thermal Analysis

Data Station. The DSC was calibrated using both indium and potassium chromate standards. The resin samples were heated from 300°K to 500°K at 20 deg/min. Ice water was used as the coolant, and the nitrogen purge gas was set to give a flow of 20 mL/min over the sample. A complete detailed working description of the Differential Scanning Calorimeter is provided in a previous document [5].

#### 2.4. $T_g$ and $T_{min}$

The Thermal Analysis Data station uses the output from the DSC to calculate several different parameters, such as the glass transition temperature ( $T_g$ ) and the peak minimum temperature ( $T_{min}$ ). (By convention the peak inflection point, which is maximum in the endo direction, is designated as the peak minimum.) Glass transition temperatures were only obtainable for certain resin mixtures. For the majority of the resins evaluated the  $T_g$  peaks were immediately followed by an exothermic cure peak, which prevented a precise glass transition analysis of those resins. The  $T_{min}$  values, however, were available for all of the polyester and vinyl ester resin mixtures. The  $T_{min}$  values are generally only several degrees higher than the  $T_g$  values and thus these values were used to demonstrate the effect of post-curing and antimony trioxide addition.

Values such as peak minimum, peak onset and heat of fusion are conventionally calculated by choosing the limits as points just before and just after the recorded peak; however, peak minimum values calculated by the data station are affected by the flatness of the peak baseline (the line drawn between the two selected points on each side of the peak). The computer sets the  $T_{min}$  value as the point with the greatest perpendicular distance from the peak baseline. Usually, in a glass transition thermogram, the baseline after the peak is higher than the baseline before the peak, and thus the peak baseline slopes upwards. This lowers the  $T_{min}$

value as selected by the computer. This principle is demonstrated in Figure 1, in which the  $T_{\min}$  value calculated using a sloping baseline is 0.8 degrees lower than that when a horizontal baseline was forced. Therefore, the peak minimum values must be obtained by choosing two limits inside the peak that will produce a horizontal baseline. This procedure was used to obtain the peak minimum values reported in Table 1.

### 3. RESULTS AND DISCUSSION

Fox [6] has shown that the  $T_g$  is related to the cross-link density of a thermoset through the following equation.

$$T_g = T_g \text{ (copolymerization)} + (\Delta T_g)MW + KU$$

where  $T_g \text{ (copolymerization)}$  = the change in  $T_g$  as the oligomeric polymer incorporates the cross-linker into its structure,

$(\Delta T_g)MW$  = the increase in  $T_g$  due to the increase in molecular weight of the prepolymer which undergoes chain extension prior to gelation, and

$KU$  = effective cross-link density.

The first two terms on the right hand side of the equation are constant after gelation because the material is considered to have an infinite molecular weight due to its three dimensional network structure. In other words,  $T_g$  is proportional to the cross-link density ( $U$ ) after gelation.

The  $T_g$  of a polymer is not an absolute number because it depends on the technique used to make the measurement (DSC, DTA or DMA) as well as the selected test parameters. However, the  $T_g$ 's measured under identical conditions can be compared in order to describe the molecular internal structure of the material.

Figures 2 through 6 show the combined DSC plots for each of the individual resin system studied at the various different cure temperatures. Examination of the DSC thermograms for Hetron 27196 obtained after a one hour post-cure at 50°C, 75°C and 100°C (Curves b-d, Figure 2) demonstrate the observed increase in peak minimum location and the corresponding change in the total peak shape, as a function of increasing post-cure temperature. The presence of an exothermic peak, which follows the endothermic glass transition peak in the thermograms, is indicative of incomplete curing of this sample when it was cured at ambient temperature (curve a). The exothermic peak reduced in size as the post-cure temperature of the resin was increased, until it was no longer observed (curve d). The DSC thermograms for the other four resin systems (Figures 3-6) exhibit these same trends, albeit to a lesser extent.

It is interesting to note that two of the resin systems, Hetron 99P and Hetron 692TP25 (Figures 4 and 5), have a small increase (5-15°C) in the measured  $T_{min}$  value as a function of the increasing cure, while the other three resin systems, Hetron 197AT, Hetron 27196 and Derakane 510A (Figures 2, 3 and 6), have a larger increase (60-75°C) in the measured  $T_{min}$  value (Table 1). This may be related to the degree-of-cure in the initial room temperature curing process.

According to Fox's equation, once the resin is hardened any observed increase in  $T_g$  should be attributed to an increase in the cross-link density and not to an increase in the molecular weight (MW). To confirm this, the five polymers were subjected to MW determinations using high performance size exclusion chromatography (HPSEC).

Figure 7 shows a plot of HPSEC retention time, which is related to the molecular weight, vs. post-cure temperature for the five resin systems. As can be seen from the data,

the relative molecular weights of the resins did not seem to increase to any notable extent as the post-cure temperature was increased; however, the observed increase in the  $T_{min}$  values and the disappearance of the curing exotherm in the corresponding DSC thermograms indicates that, for each given thermoset resin, the post-curing resulted in a greater cross-link density ( $\nu$ ) and therefore a higher degree-of-cure.

Since the  $T_g$  of a polymer is closely related to the physical properties of the polymer, it is reasonable to assume then that the resin systems with the least amount of change to their glass transition point, as a function of post-curing, would be the polymers with a minimal amount of change to their physical properties. However, since increasing the degree-of-cure of a thermosetting polymer generally increases the fire retardancy, then the resin system which attained the highest  $T_{min}$  value upon curing would be the better fire resistant resin. It must be pointed out that for several of the resin systems studied, these higher  $T_g$ 's were only achieved by post-curing the resins at 100°C for one hour.

The effect of the degree-of-cure on the mechanical properties of a polyester resin system is demonstrated in Figure 8. A plot of the measured storage modulus vs. temperature for a series of increasing degree-of-cure samples (as reflected by increased post-cure temperatures) indicated that for samples which were only partially cured ( $T_{post-cure} < 75^\circ\text{C}$ ), the storage modulus decreased in the temperature range of 40-80°C; however, for the samples which were more completely cured ( $T_{post-cure} > 75^\circ\text{C}$ ), the storage modulus remained relatively constant in the same temperature range. The ability of a material to exhibit a minimal change to its modulus over a wide temperature range is an important requirement for structural applications.

An earlier study indicated that fire-retardants such as antimony trioxide can be added to these polyester and vinyl ester resin systems to impart improved fire performance [7].. Consequently, the effect that addition of antimony trioxide has on the glass transition temperature and cross-link density was investigated.

Using the Differential Scanning Calorimeter, values for  $T_{min}$  were obtained for the same five resins with 0, 2.5, 5, 7.5 and 10 phr antimony trioxide loadings. The plots of  $T_{min}$  vs post-cure temperature are shown in Figures 9 through 13, and it can be noted that the  $T_{min}$  values increased with increased post-cure temperature for all of the resin systems irrespective of the amount of antimony trioxide added. Although for some of the resins the data were somewhat scattered, a trend can easily be seen which suggests that antimony trioxide additions have a negligible effect on the measured  $T_{min}$  values for these resins. That is to say, the addition of up to 10 phr antimony trioxide had no measurable effect on the degree-of-cure (cross-link density) as a function of post-cure temperature, and thus would have no effect on the net mechanical properties.

#### 4. CONCLUSIONS

Results indicated that the effect of post-curing was to increase the amount of cross-linking for the polyester and vinyl ester resins, as evident by an increase in the glass transition temperature. The increased degree-of-cure resulted in a beneficial change to the physical properties as reflected by the invariance in the storage modulus; however, the addition of up to 10 parts per hundred resin (phr) of the fire-retardant, antimony trioxide was shown to have a negligible effect on the onset of the glass transition point.

**TABLE 1**

Peak Minimum Values by DSC as a Function of Post-Cure  
Temperature for Resins with no Antimony Trioxide

Resin	Post-Cure Temperature (°C)				
	20	50	75	100	125
Hetron 99P	73.63	75.01	-	76.33	79.39
Hetron 197AT	75.35	78.75	-	119.87	139.63
Hetron 27196	59.71	74.73	98.25	113.75	-
Hetron 692TP25	57.43	57.06	66.20	67.17	-
Derakane 510A	53.90	51.28	-	111.36	131.46



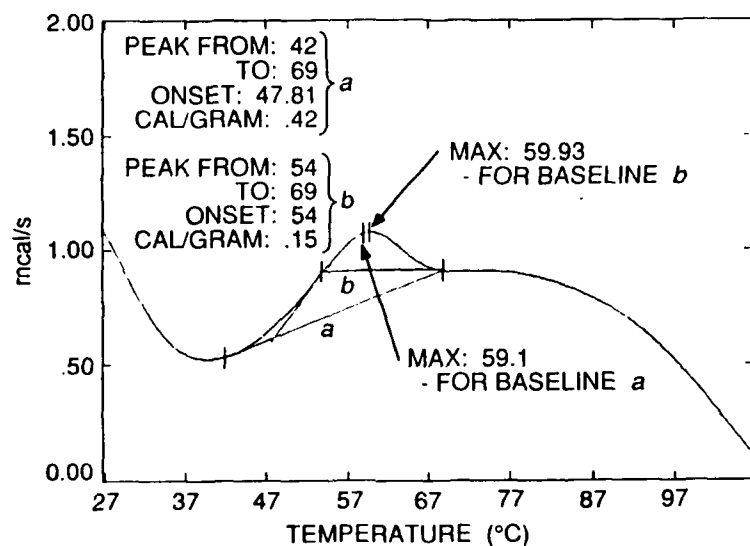


Figure 1 DSC Thermogram demonstrating the difference in  $T_{min}$  values as a function of peak limits.

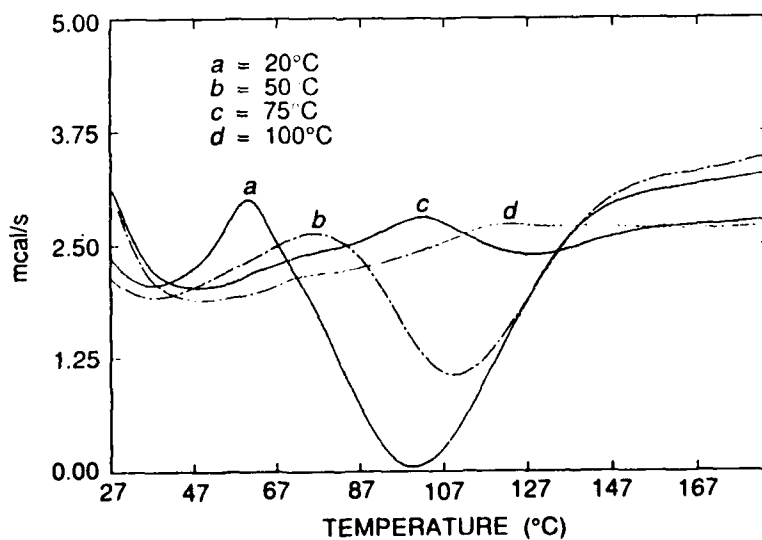


Figure 2 DSC Thermograms vs. Cure Temperature for Hetron 27196.

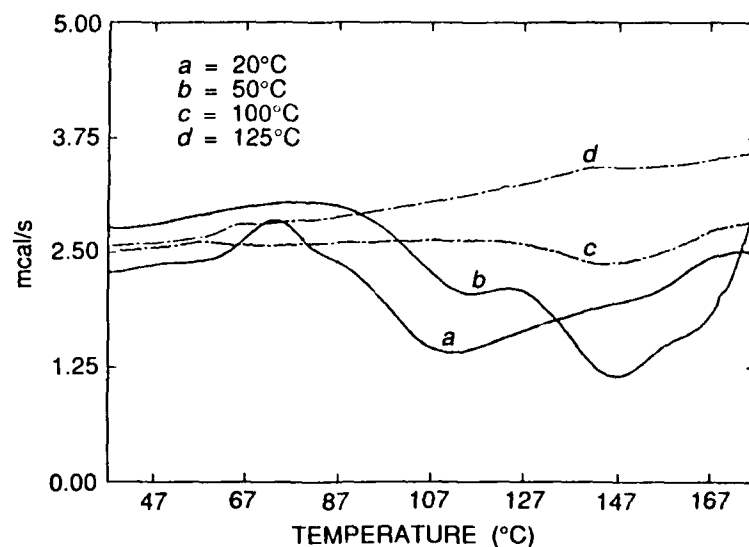


Figure 3 DSC Thermograms vs. Cure Temperature for Hetron 197 AT.

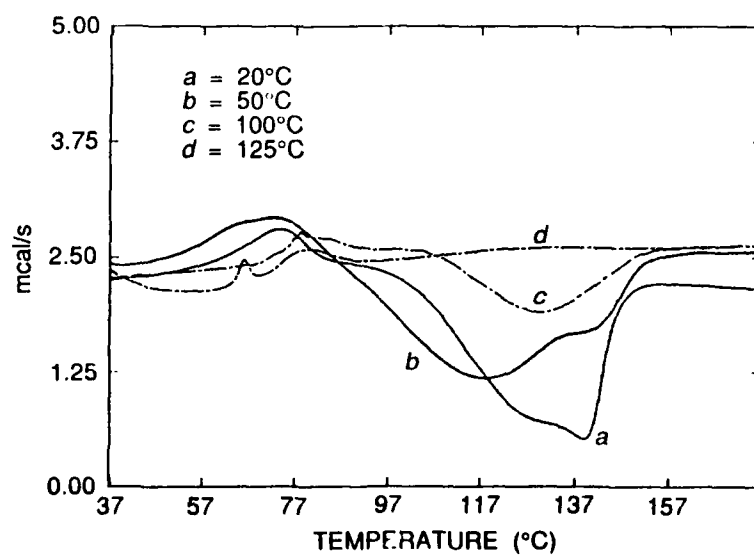


Figure 4 DSC Thermograms vs. Cure Temperature for Hetron 99P.

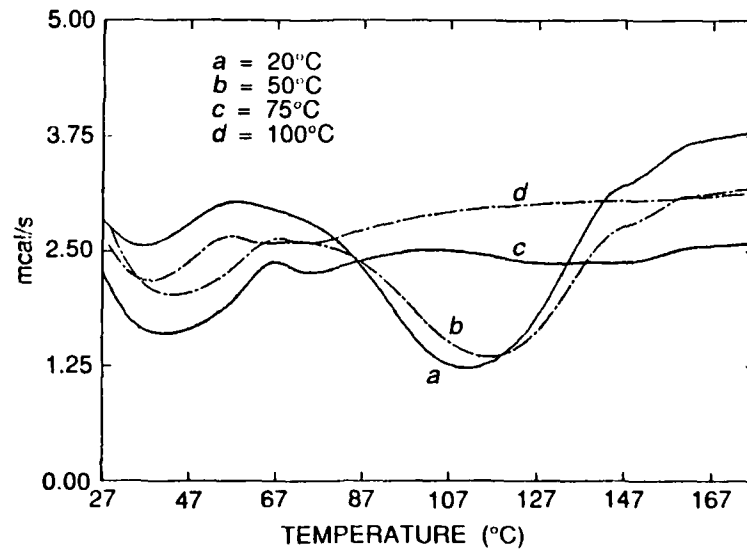


Figure 5 DSC Thermograms vs. Cure Temperature for  
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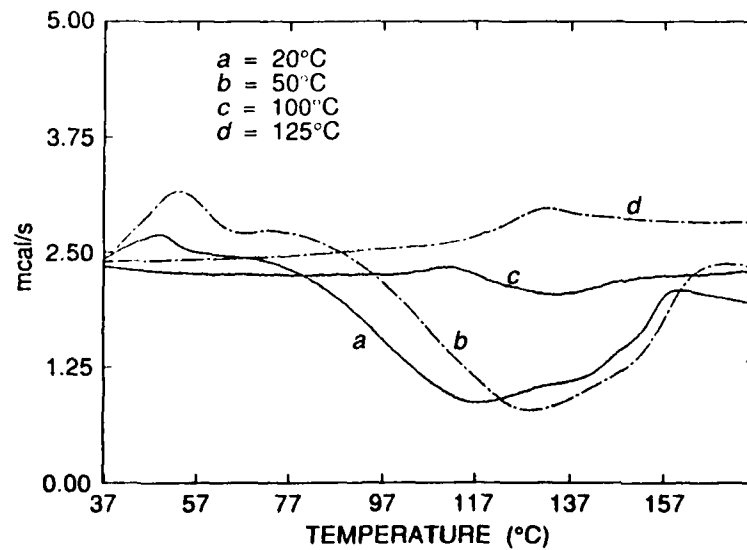


Figure 6 DSC Thermograms vs. Cure Temperature for  
Derakane 510A.

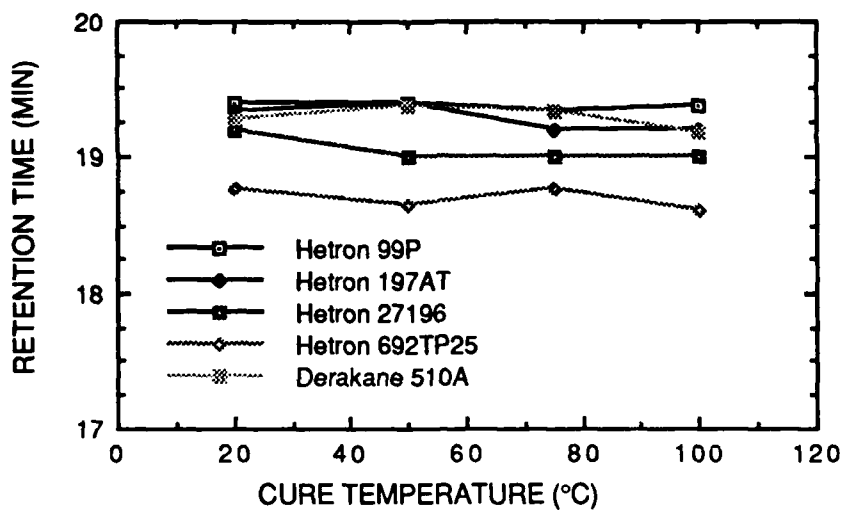


Figure 7 Plot of Retention Time for Gel Permeation Chromatography vs. Cure Temperature for all Resins.

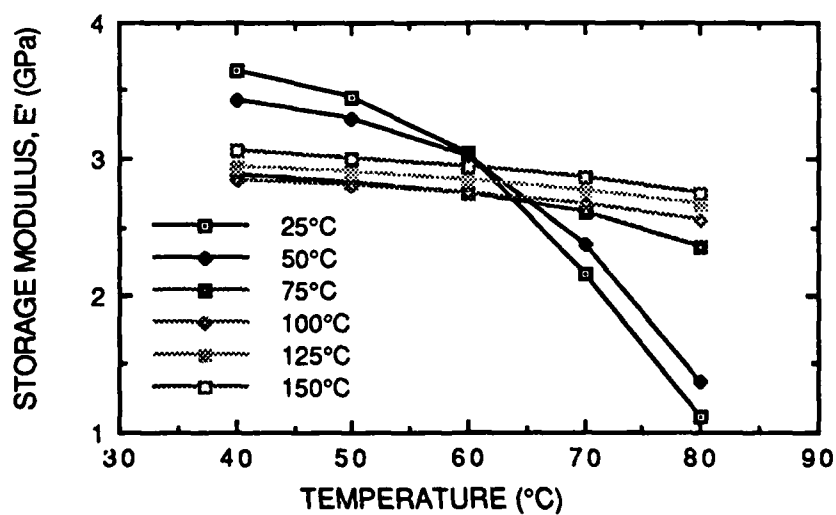


Figure 8 Plot of Storage Modulus vs. Temperature for Hetron 27196 as a Function of Post-Cure Temperature.

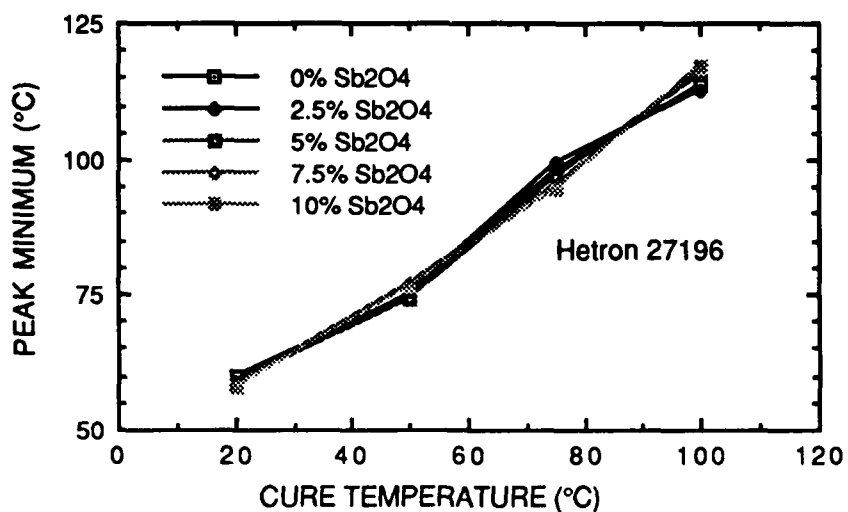


Figure 9 Plot of Peak Temperature vs. Cure Temperature for Hetron 27196.

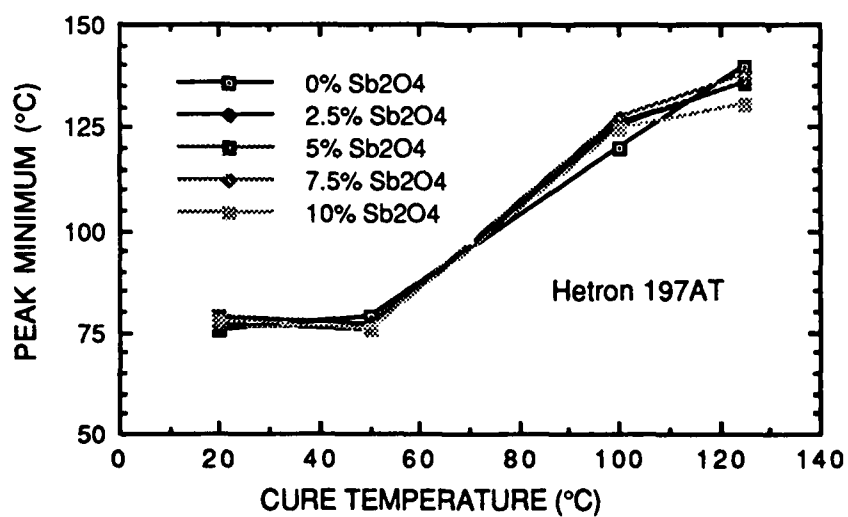


Figure 10 Plot of Peak Temperature vs. Cure Temperature for Hetron 197 AT.

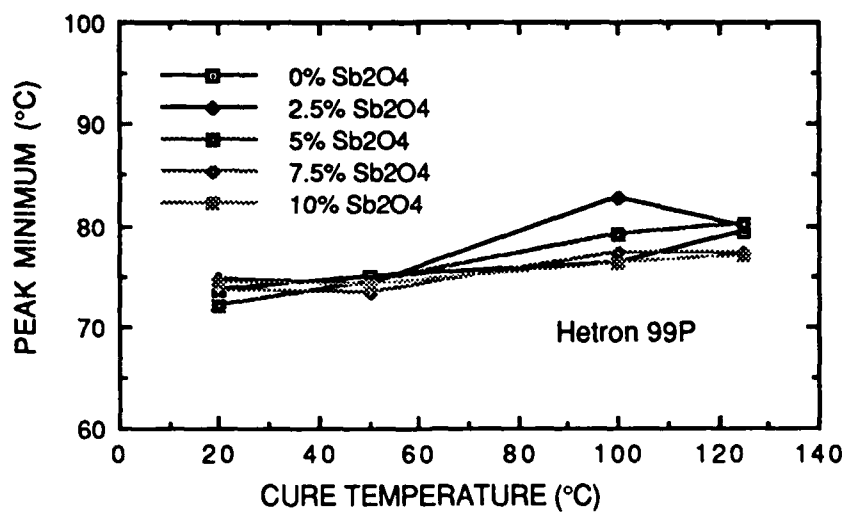


Figure 11 Plot of Peak Temperature vs. Cure Temperature for Hetron 99P.

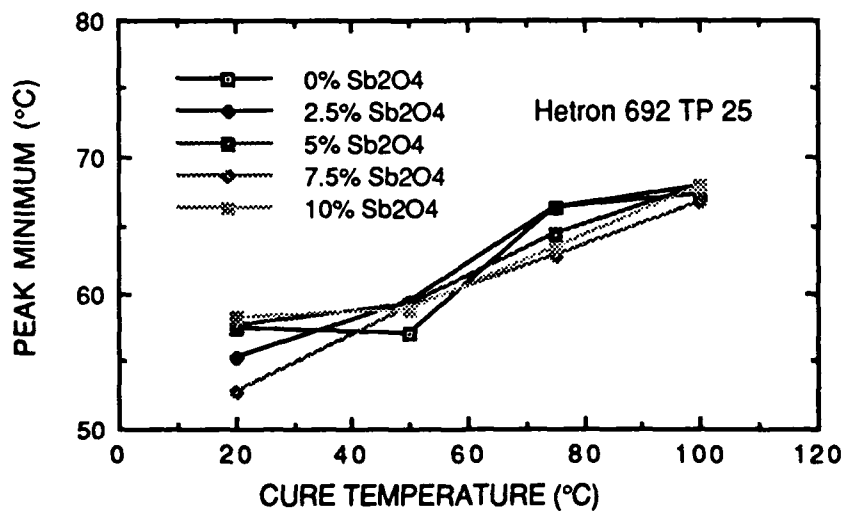


Figure 12 Plot of Peak Temperature vs. Cure Temperature for Hetron 692 TP 25.

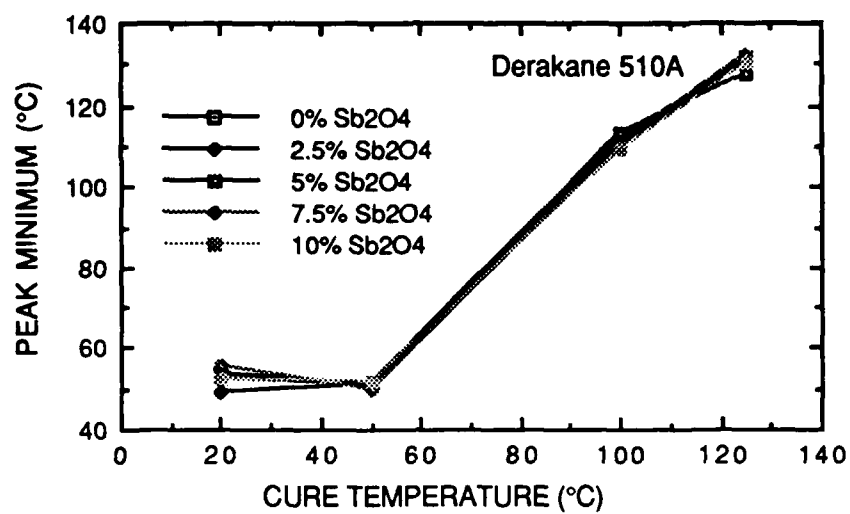


Figure 13 Plot of Peak Temperature vs. Cure Temperature for Derakane 510A.

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The effect of post-curing on the glass transition temperature ( $T_g$ ) of a variety of styrene cross-linked polyester and vinyl ester resins was determined by monitoring the changes to  $T_g$  using a Differential Scanning Calorimeter. The effect of adding the inorganic fire-retardant, antimony trioxide, on the  $T_g$  of these resin systems was also investigated.

Results indicated that the effect of post-curing was to increase the amount of cross-linking of the polyester and vinyl ester resins, as evident by the beneficial increase in the glass transition temperature and changes to the storage modulus. The addition of up to 10 parts per hundred resin (phr) of the fire-retardant, antimony trioxide, had a negligible effect on the onset of the glass transition point.

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polyester  
vinyl ester  
post-cure  
glass transition  
antimony trioxide  
DSC

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